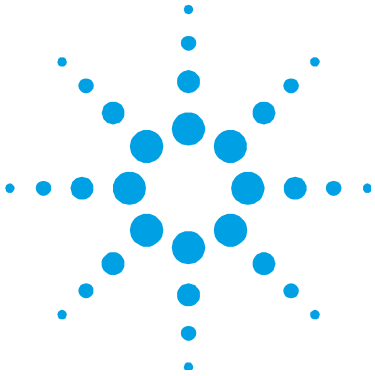

Agilent Vapor Generation Accessory VGA 77 and VGA 77P

User's Guide

Agilent Technologies

Notices

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Safety Notices

CAUTION

A **CAUTION** notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in damage to the product or loss of important data. Do not proceed beyond a **CAUTION** notice until the indicated conditions are fully understood and met.

WARNING

A WARNING notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in personal injury or death. Do not proceed beyond a WARNING notice until the indicated conditions are fully understood and met.

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1. Safety Practices and Hazards

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General

Operation of the VGA 77 with an Atomic Absorption spectrometer (AA) or VGA 77P with an Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES) involves the use of compressed gases and hazardous materials, including corrosive fluids. Unskilled, improper, or careless use of equipment can create explosion hazards, fire hazards or other hazards which can cause death, serious injury to personnel, or severe damage to equipment and property.

Appropriate safety practices have been included in this operation manual and your spectrometer operation manual, to help users operate the equipment safely. Read all safety practices thoroughly before attempting to operate your system.

If the equipment is used in a manner not specified by the manufacturer, the protection provided by the equipment may be impaired.

Do not position the equipment so that it is difficult to operate the disconnecting device.

Hazard Warnings

In addition to the hazard warnings contained in your spectrometer operation manual, specific hazard warnings have been included in this operation manual. These warnings state the hazard, describe how to avoid it and specify the possible consequences of not following the instructions. Read all warnings carefully and observe them at all times.

Electrical Hazards

The VGA 77 contains electrical circuits, devices and components operating at dangerous voltages. Contact with these circuits, devices and components can cause death, serious injury or painful electric shock. Covers that are retained by screws on the VGA 77 may be opened only by Agilent field service engineers.

Use of the wrong supply voltage, connection of the accessory to an incorrectly wired supply outlet, or lack of proper electrical grounding can create a fire or shock hazard that can cause death, serious injury, or serious damage to equipment.

- Always use a 3-wire outlet with a ground connection that is adequately rated for the load.
- The installation must comply with local, state and national safety regulations.
- Before connecting the VGA 77, make sure the voltage selector is correctly set for the mains supply to which you are connecting the accessory (see Figure 2 on Page 23).

A blown fuse should be replaced with one of the same size and rating stated in the text near the fuse holder.

Chemical Hazards

Vapor generation methods of analysis involve the generation of toxic hydrides and use materials which are toxic, highly corrosive or otherwise hazardous. Careless, improper, or unskilled use of such materials can cause serious personal injury.

Always ensure that laboratory safety practices governing the use, handling and disposal of such materials are strictly observed. These safety practices should include the wearing of appropriate safety clothing.

In the VGA 77 system, highly concentrated acids are pumped under pressure. If a leak occurs, acid could be sprayed from the system, causing serious personal injury. Always wear approved safety glasses warranted to protect the eyes.

Never pump concentrated sulfuric acid through the VGA 77 system.

Warning and Caution Messages

A 'Note' is used to give advice or information.

The following is a list of symbols that appear in conjunction with warnings in this manual and on the spectrometer. The hazard they describe is also shown. The beginning of the warning text is noted by a warning icon:

WARNING

The following symbol may be used on warning labels attached to the Agilent VGA 77. When you see this symbol, refer to the relevant operation or service manual for the correct procedure referred to by that warning label.

A triangular symbol indicates a warning. The meanings of the symbols that may appear alongside warnings in the documentation or on the instrument itself are as follows:

Corrosive liquid

Electrical shock

Explosion hazard

Eye hazard

Fire hazard

Heavy weight (danger to feet)

Heavy weight (danger to hands)

Hot surface

Moving parts

Noxious gas

Sharp object

Information Symbols

I	Mains power on
O	Mains power off
	Fuse
	Single phase alternating current

Color Coding

The various indicator lights appearing on the instrument and any associated accessories have been color coded to represent the status of the instrument or accessory:

- A green light indicates the instrument is in normal/standby condition.
- An orange light indicates that a potential hazard is present.
- A blue light indicates that operator intervention is required.
- A red light warns of danger or an emergency.

CAUTION Respond quickly to no or low gas pressure or flow errors indicated by the blue light. Failure to do so can damage the VGA due to acid or vapor ingression caused by insufficient gas supply flow.

Electromagnetic Compatibility

EN55011/CISPR11

Group 1 ISM equipment: group 1 contains all ISM equipment in which there is intentionally generated and/or used conductively coupled radio- frequency energy which is necessary for the internal functioning of the equipment itself.

Class A equipment is equipment suitable for use in all establishments other than domestic and those directly connected to a low voltage power supply network which supplies buildings used for domestic purposes.

This device complies with the requirements of CISPR11, Group 1, Class A as radiation professional equipment. Therefore, there may be potential difficulties in ensuring electromagnetic compatibility in other environments, due to conducted as well as radiated disturbances.

Operation is subject to the following two conditions:

1 This device may not cause harmful interference.

2 This device must accept any interference received, including interference that may cause undesired operation.

If this equipment does cause harmful interference to radio or television reception, which can be determined by turning the equipment off and on, the user is encouraged to try one or more of the following measures:

1 Relocate the radio or antenna.

2 Move the device away from the radio or television.

3 Plug the device into a different electrical outlet, so that the device and the radio or television are on separate electrical circuits.

4 Make sure that all peripheral devices are also certified.

5 Make sure that appropriate cables are used to connect the device to peripheral equipment.

6 Consult your equipment dealer, Agilent Technologies, or an experienced technician for assistance.

Changes or modifications not expressly approved by Agilent Technologies could void the user's authority to operate the equipment.

2. Introduction

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About the VGA 77 and VGA 77P

The Vapor Generation Accessory 77 (VGA 77) is a vapor generation system for Atomic Absorption (AA) and the VGA 77P is for Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES) applications. Unless otherwise specified, both the VGA 77 and VGA 77P will be refered to as VGA 77 throughout the rest of this User's Guide.

Vapor generation is an extremely sensitive procedure used for determining the level of mercury, arsenic, selenium and a range of other hydride-forming elements in samples. The sample capillary is placed in the solution to be measured and the VGA 77 pumps the sample through a reaction coil where it is automatically acidified and mixed with a suitable reductant. The resulting vapor is transferred to an atomization cell with AAS or to the plasma for ICP-OES for determination by the spectrometer.

The VGA 77 is supplied in a modular form, with a pump unit and separate reagent module. Since ICP and AA applications use different gas/liquid separators, using separate plumbing assemblies for different reagents enables:

- Flexibility—the VGA 77 can be used with both AAs and ICPs
- Quick and simple change of applications, for example from AA to ICP or between different types of analyses

- Cleaner operation—there is no chance of cross-contamination between applications
- Ease of operation—there is no need to clean reagent bottles and tubing between applications as each application has a dedicated reagent module. Similarly, you can change from AA to ICP applications by changing reagent modules

NOTE Part numbers for additional reagent modules are listed on Page 79.

About this Manual

This manual includes instructions for installing, using and maintaining the VGA 77. You should use this manual in conjunction with your spectrometer operation manual and any other component or accessory manuals (for example a printer or sampler).

As you use this manual, there are some procedures that will apply to specific applications only. For example, the section referring to 'Installing the Cell' does not apply if you are using your VGA 77 with an Agilent ICP-OES system. The instructions will inform you when this occurs.

NOTE This manual contains basic information about using the VGA 77 with ICP-OES and AA instruments. For detailed information, refer to the ICP Expert, ICP Expert II or SpectrAA Help.

Specifications

Environmental

Your accessory is designed for indoor use only. It is suitable for the following categories:

- Installation Category II
- Pollution Degree 2
- Equipment Class I

Condition	Altitude	Temp (°C)	Humidity (%RH) non-condensing
Non-operating (transport)	0-2133 m (0-7000 ft)	5-45	20-80
Operating within performance specifications	0-853 m (0-2800 ft) 853-2133m (2800-7000 ft)	10-35 10-25	8-80

Power

If necessary, replace the power cord only with a cord equivalent to the one specified in the site preparation guide.

Information is correct at time of printing—information on the back of the accessory is the most up-to-date.

Voltage	100, 120, 220, or 240 volts AC
Frequency	50-60 Hz
Consumption	approx. 20 VA

Connections

Mains inlet coupler: 6 A 250 VAC IEC type

Mains power cord connector:

Australia	10 A 250 VAC	Complies with AS3112
USA	10 A 125 VAC	Complies with NEMA 5-15P
Europe	6 A 250 VAC	Complies with CEE7 sheet vii or NFC61.303VA

Gas Supplies

Argon or nitrogen, 99.99% pure (minimum)

NOTE Only argon gas can be used when the VGA 77 is coupled with an ICP spectrometer.

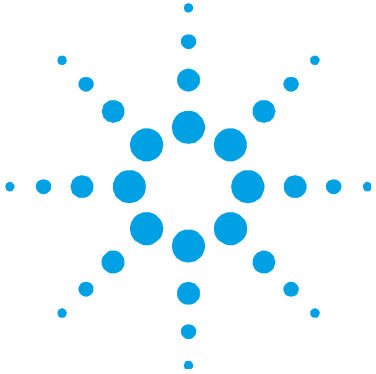
The inert gas must be dry and dust-free, otherwise you must insert a suitable filter in the supply line.

Permissible pressure range	300 to 400 kPa (43 to 57 psi)
Recommended pressure	350 kPa (50 psi)
Nominal flow rate	90 mL/minute

Gas connections, inert gas inlet: 6 mm (1/4 in) ID reinforced PVC hose 3 meters (10 ft) long

Weights and Dimensions

System unit	Width	Depth	Height	Weight
VGA 77 for AA, Vista Series ICP-OES and 5100 ICP-OES	320 mm 13 in.	210 mm 8 in.	270 mm 11 in.	5.5 kg 12 lb
Shipping dimensions	590 mm 23 in.	475 mm 19 in.	320 mm 13 in.	11 kg 24 lb
VGA 77 for 700 Series ICP-OES (with bracket)	385 mm 13 in.	340 mm 14 in.	195 mm 8 in.	6 kg 13 lb
Shipping dimensions	590 mm 23 in.	490 mm 19 in.	320 mm 13 in.	10 kg 22 lb



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Unpacking

Carefully open the shipping case and the enclosed packages. Inspect all parts for damage in transit. Any damage should be reported immediately.

You should have received the following:

- VGA 77 pump unit (with mounting bracket if ordered for the 700 Series ICP-OES instruments)
- Cover for the pump unit
- Drip tray for the pump unit

- Reagent module with two reagent bottles
- Two quartz cells with a length of black fluoro-elastomer tubing (AA only)
- Power cable
- Tubing and connectors kit and spare 6 mm ID hose (used to connect a mercury trap)
- Clear tubing (10 mm ID)
- Hose clamp and fuses
- Cell holder for Mark 7 burner (AA only)

NOTE Spare parts and their part numbers are given on Page 79.

Installation

Before proceeding with the installation, you should read the 'Safety practices and hazards' section at the start of this manual. You should also be completely familiar with the safety procedures required for operation of your spectrometer.

During installation, refer to the following diagram to help you identify the different parts of your VGA and pump module.

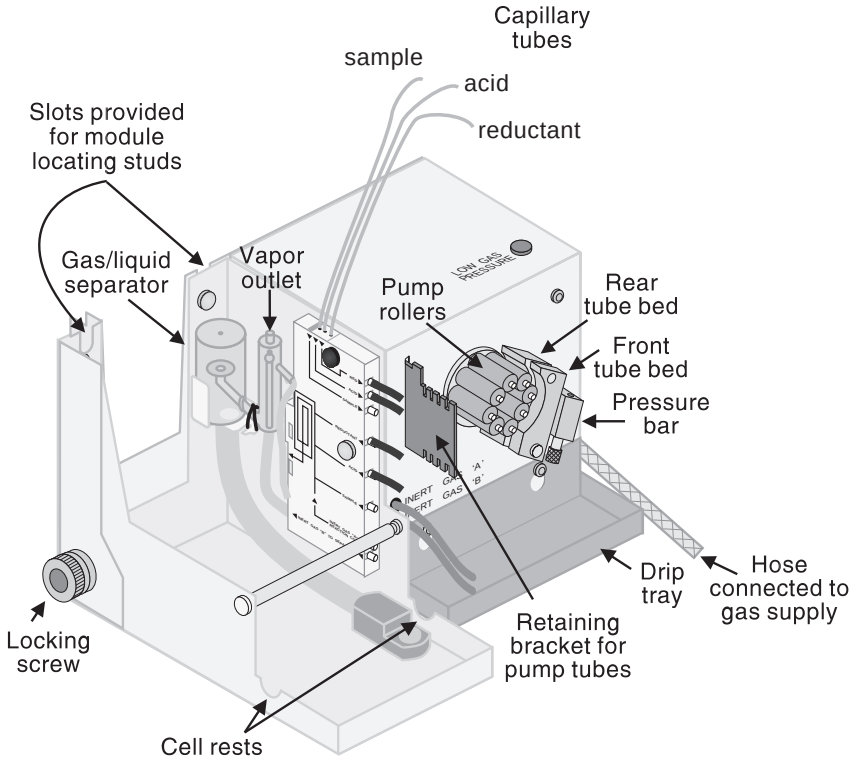


Figure 1. VGA 77 with AA module installed

Installation Checklist

Use the following checklist to help you complete each step of the installation.

- Check that the correct gas supply is available and then connect your VGA 77 to the supply. See Page 20.
- Check that the correct power supply is available, the correct fuses are installed and that the voltage selector is correctly set. See Page 22.
- Check the environmental requirements. See Page 15.
- Install the pump unit. See Page 24.
- Install the reagent module. See Page 24.

- Install the drain tubing. See Page 25.
- Install the VGA pump tubing. See Page 27.
- Install the burner clamps (AA only). See Page 29.
- Install the cell (AA only). See Page 36.
- Connect the gas/liquid separator outlet to:
  the inlet of the absorption cell (AA), or
  the sample introduction system (ICP).
  See Page 38.
- If necessary, connect a mercury trap to the cell exhaust to comply with local regulations on the discharge of mercury vapor (AA only). See Page 41.

Gas Supply Requirements

A regulated supply of argon must be provided for your VGA 77 through the captive hose located in the right side panel. You may also use nitrogen with the AA reagent module only.

NOTE Nitrogen must not be used with the ICP module as it may degrade the performance of the plasma.

The gas supply must comply with the specifications in Section 2. The VGA is fitted with a 6 mm (1/4 in.) internal diameter reinforced plastic hose for connection to a standard barbed-tail connector. If you are in any doubt about the correct fittings to use, consult your supplier.

Connecting the Gas Supply

To connect the gas supply:

1 Place the clamp over the free end of the captive hose and connect the free end of the captive hose to the metal barbed-tail connector on the gas supply or to your gas supply regulator.

2 Tighten the clamp, by adjusting the screw, to secure the gas supply hose to the gas supply. Adjust the pressure at the regulator to the recommended pressure.

CAUTION The argon supply pressure supplied to the VGA 77 must be 300 to 400 kPa (43 to 57 psi). The 400–600 kPa (57–88 psi) pressure required to supply argon to the ICP spectrometer is too high for direct connection to the VGA 77.

TIP The VGA 77 gas supply may be taken from the main argon gas line to ICP spectrometers if you use the following precautions:

1 Insert a suitable 'T' piece into the main argon gas supply to divert some of the gas to the VGA 77.
2 Attach a regulator to control the flow of argon to the VGA 77.
3 Place a tap in the argon supply to the VGA 77. The tap is required to shut off the argon supply to the vapor generator, as gas can flow through the VGA 77 even when it is switched off.

Within the VGA 77, the inert gas supply is divided into two branches. The first branch is controlled by a solenoid stop valve. It supplies gas to the inlet side of the reaction coil. The second branch supplies inert gas directly through the gas/liquid separator.

NOTE Inert gas will flow through the separator even with the VGA 77 switched off. When the VGA 77 is switched on, it will flow through both branches. To reduce gas consumption when the VGA 77 is not in use, a shut-off valve in the inlet gas supply is recommended. The gas supply can then be shut-off when the VGA 77 is switched off.

During operation, the gas supply pressure is automatically monitored by a built-in sensor. If the gas supply is interrupted, or if the supply pressure falls below the specified minimum, the blue indicator light will be illuminated, and the gas supply to the reaction coil is shut off.

The indicator light is the single blue light on the top panel of the accessory. If the indicator light turns on, switch the VGA 77 off immediately, rectify the gas supply problem, then switch the VGA 77 back on. If the indicator light again turns on, you should call your Agilent field service engineer.

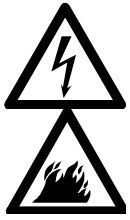
CAUTION Respond quickly to no or low gas pressure or flow errors indicated by the blue light. Failure to do so can damage the VGA due to acid or vapor ingression caused by insufficient gas supply flow.

Power Supply Requirements

The voltage selector, mains receptacle, power switch and fuses are located on the right-hand side of the accessory.

Before connecting the mains power for the first time, check that the correct power supply is available, the voltage selector switches on the accessory are set correctly, and that the correct fuses are installed and both fuses are of the same rating.

WARNING



Shock and Fire Hazard
Application of the wrong supply voltage, connection of the accessory to an incorrectly wired supply outlet, or lack of proper electrical grounding can create a fire or shock hazard that can cause death, serious injury or serious damage to equipment.

For power supply and fuse specifications refer to the ‘Specifications’ section in Section 2 of this manual. Refer to the ‘Fuses’ section in the Maintenance chapter of this manual for details of how to inspect and replace fuses.

Selecting the Voltage

Refer to the table immediately above the selector switches or consult the following table and set the switches as required. For example, if the accessory is connected to 240 volts, the table tells you that the switch setting should be 'AD'. This means the left switch should be up (position 'A') and the right switch should be down (position 'D').

Voltage	Switch setting	Switch positions	
		Left	Right
240 ± 10%	AD	Up	Down
230 +14% -6%	AD	Up	Down
230 +6% -14%	BD	Down	Down
220 ± 10%	BD	Down	Down
120 ± 10%	AC	Up	Up
100 ± 10%	BC	Down	Up

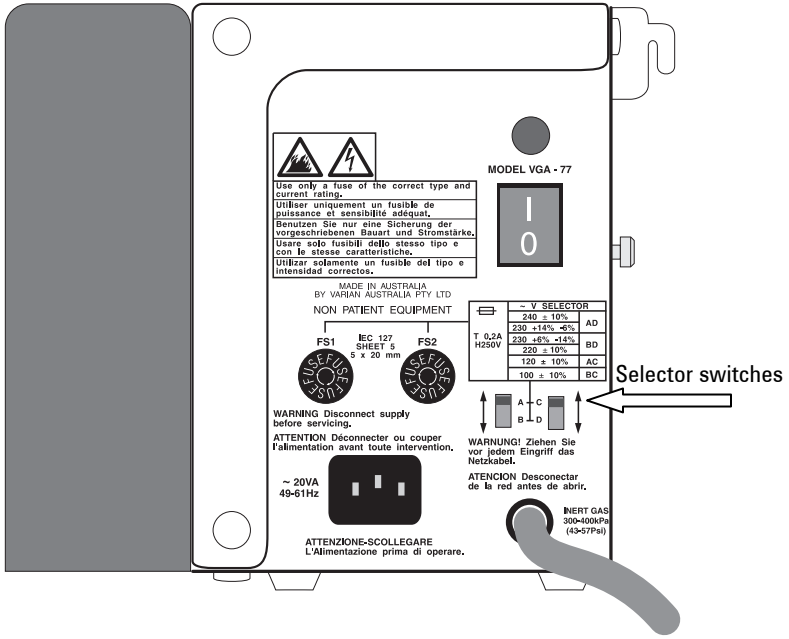


Figure 2. Side of the VGA

Installing the Pump Unit

WARNING

Moving parts

The peristaltic pump has moving parts that can cause damage to hands or fingers if caught underneath the clamps during operation. Keep hands away from the peristaltic pump while it is running.

To install the VGA 77 on an Agilent AA:

Insert the hooks on the back of the unit into the slots located on either side of the Agilent AA sample compartment.

To install the VGA 77 on an Agilent 700 Series ICP-OES instrument:

1. Insert the four stainless steel thumbscrews into the four holes towards the front of the instrument sample compartment (two on each side).
2. Hook the four grooves in the VGA 77 mounting bracket over the four thumbscrews in the sample compartment.

For an instrument other than an Agilent AA or Agilent 700 Series ICP-OES, you should place the VGA 77 flat on a bench, close to the sample compartment of the instrument.

When the VGA 77 is in position, fit the drip tray to the front of the pump unit by positioning the large holes over the studs on the front of the VGA 77 and sliding the tray downwards.

Installing a Reagent Module

To install a reagent module into the VGA 77:

1. Loosen the locking screw at the lower rear left hand corner of the pump unit.
2. Lower the reagent module into the space adjacent to the left side of the pump unit, ensuring the reagent module locating studs are fully home in the slots.
3. Wind the locking screw in until it holds the module firmly in place.

4 Fit the end of the tube from the pump unit marked ‘Inert gas ‘A’’, (the top tube on the front of the VGA), to the nipple near the bottom of the reagent manifold marked ‘Inert gas ‘A’ to reaction coil’. The nipple has a contoured profile. Push the tubing onto the nipple until it covers all of the contoured section.

Figure 3. Inert gas tubing from pump unit correctly connected to nipples on reagent manifold

5 Fit the end of the tube from the pump unit marked ‘Inert gas ‘B’’ to the nipple near the bottom of the reagent manifold marked ‘Inert gas ‘B’ to separator’. The nipple has a contoured profile. Push the tubing onto the nipple until it covers all of the contoured section.

NOTE To remove a module and replace it with another, see instructions on Page 54.

Installing the Drain Tubing

WARNING

Corrosive Liquid
Waste solutions from the VGA 77 may contain concentrated acids which can cause severe burns. Your waste vessel must be of durable, acid-resistant material. Do not use a glass container. Locate the vessel where it cannot be knocked over. Empty it frequently. Dispose of waste solutions in accordance with relevant safety practices.

AA

There are two drain outlets located on the underneath side of the reagent module. The outlet closest to the front face of the module is for tray overflow, the other is to drain liquid from the gas/liquid separator. Drain tubing should be connected to both of these drain outlets.

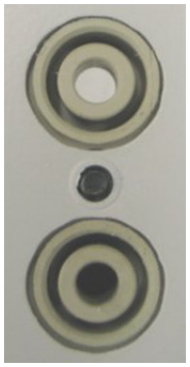


Figure 4. Drain outlets

To connect drain tubing to the drain outlet of an AA reagent module:

1. Cut a suitable length of the plastic drain tubing supplied with your reagent module.
2. Connect one end of the tubing to the large outlet underneath the module tray. The tubing should be connected between the inner and outer rings on the outlet.
3. Place the free end in your waste vessel. Ensure that the tubing is free of kinks or sharp bends so that the waste liquid will drain freely into the waste vessel.

NOTE The end of the tube must be above the surface of the liquid in the waste vessel. If the end of the drain tube is submerged, possible back-pressure may lead to unreliable results.

ICP

To connect drain tubing to the drain outlet of an ICP reagent module:

1. Fit a purple-black pump tube to the instrument peristaltic pump as described in your instrument operation manual.
2. Connect the inlet of the purple-black pump tube to the drain outlet of the gas/liquid separator in the ICP reagent module. (Refer to Page 41 for a diagram of the ICP plasma gas/liquid separator.)
3. Attach a suitable length of the PVC tubing supplied to the outlet of the purple-black pump tube from the ICP instrument.
4. Place the free end of the PVC tubing into the waste container. Ensure that the tubing is free of kinks or sharp bends so that the waste liquid will drain freely into the waste vessel.

NOTE Use only the purple-black tubing for draining the ICP/VGA 77 system. Other smaller diameter pump tubing, for example the black-black tubing, may not give a large enough drain flow rate. The instrument peristaltic pump rate must be sufficient to remove all waste liquid from the gas/liquid separator (typically 40–45 rpm).

Installing the VGA 77 Pump Tubing

To install the VGA-77 pump tubing:

1. Open the pressure bar to release the front and rear tube beds. Swing both tube beds clear of the pump rollers.
2. Fit black-black tubing into the two innermost tubing slots and purple-black tubing to the outer most tubing.
3. Connect the inlet end of the innermost tube to the black rubber connector at the top of the reagent manifold marked ‘Red. ->’. Connect the other end to the black rubber connector near the middle of the reagent manifold marked ‘Reductant <-’.

4. Connect the inlet end of the middle tube to the black rubber connector at the top of the reagent manifold marked ‘Acid ->’. Connect the other end to the black rubber connector near the middle of the reagent manifold marked ‘Acid <-’.
5. Connect the inlet end of the outer tube to the nipple at the top of the reagent manifold marked ‘Sample ->’. Connect the other end to the nipple near the middle of the reagent manifold marked ‘Sample <-’. The nipples have a contoured profile. Ensure the tube is pushed all the way home and covers all of the contoured section of the nipple.
6. Swing the rear tube bed against the rear tubes and ensure that:
   a. The bed bears evenly against the tubes
   b. The tubes are seated squarely on the pump rollers.
7. Adjust the position of the tubes if necessary.
8. Repeat Step 6 with the front tube bed.
9. Adjust the speed of the peristaltic pump on the ICP spectrometer to ensure that liquid is being pumped out of the gas/liquid separator faster than it is being pumped in by the VGA 77.

   For best results, try not to overfill the gas liquid separator and try not to completely empty it.

TIP If you shorten the ‘tails’ of the pump tubing, you may reduce reagent and sample volume in the tubing, thereby reducing sample consumption and reducing pre-read delays.

TIP The mixing coil is particularly required for mercury analysis. Analysis times for hydride elements can be reduced by using a shorter length of the mixing coil tubing. A separate module could be dedicated to this application.

Installing the Burner Clamp

This section applies to AA modules only.

CAUTION Do not use the cell with a nitrous oxide–acetylene burner. The cell will be destroyed if exposed to a nitrous oxide-acetylene flame.

Mark 7 Burner

The VGA 77 is supplied with a cell holder for the Mark 7 burner. The Mark 7 does not require mounting brackets as the cell holder simply sits on top of the burner.

Figure 5. Mark 7 burner without holder

To mount the cell holder on the Mark 7 burner:

1 Position the seat cut-outs of the holder over the back edge of the burner.

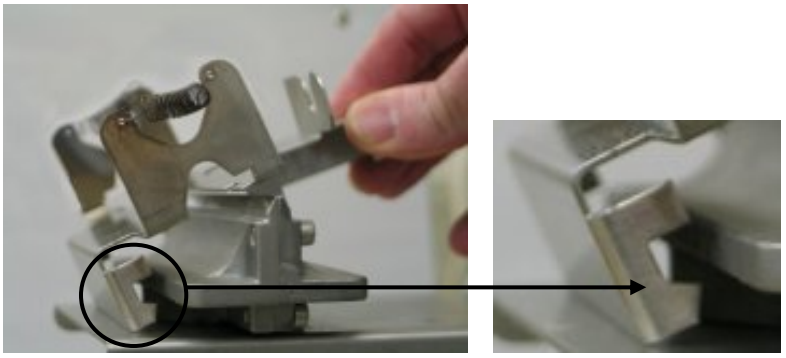


Figure 6. Close up view of the cut outs.

2 Pull the holder forward so that the cut-outs hook around the burner edge.

Figure 7. Cell holder pulled forward with cut-outs hooked around burner edge

3 Lower the holder into position.

Figure 8. Lowering the cell holder into position on the Mark 7 burner

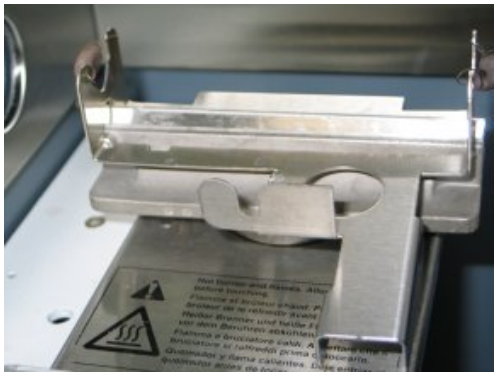


Figure 9. Mark 7 burner with holder in position

Mark VI and VI-A Burners

If you have a Mark VI or Mark VI-A burner, you will need a cell holder and suitable mounting brackets for these burners. (Refer to Page 79 for ordering information.)

To install these mounting brackets on the burner:

1. Align the burner clamps at either end of the burner, with the mounting peg pointing inward and the hole in the end of the plate aligned over the hole in the end of the burner.
2. Use the screws provided to fix the burner clamps in position.

NOTE The clamps can be left permanently installed since they will not interfere with normal flame analyses.

Figure 10. Mark VI burner with burner clamps installed

NOTE The Mark VI-A burner looks different to the Mark VI burner, however the procedure for installing the clamps is the same.

Mark V Burner

If you have a Mark V burner (or earlier version), you will need a cell holder and burner mount for this burner. (Refer to Page 79 for ordering information.)

To install the burner clamp for the Mark V burner:

1 Slacken the two clamping screws on the underside of the clamp.

NOTE The 'front' of the air-acetylene burner carries the identification label. Orient the clamp so that the two mounting pegs are at the front of the burner. This will ensure that the burner identification label is still visible through the 'open' portion of the clamp.

2 Fit the clamp over the burner so that the two positioning lugs at each end of the clamp rest on top of the burner, on either side of the burner slot.

3 Tighten the clamping screws.

Figure 11. Mark V burner with burner clamps installed

NOTE The clamp can be left permanently installed since it will not interfere with normal flame analyses.

Installation of Zeeman Adapter for 240Z/280Z AA

The VGA for Zeeman Adapter allows the VGA to be used with the Agilent 240Z/280Z AA. The adapter allows installation of the following cells in the sample compartment:

- Mercury flow-through cell
- Standard hydride absorption cell
- ETC 60 Cell Mk 2

Assembly

The following instructions outline the steps required to assemble and change cell holders of the VGA 77 for Zeeman Adapter. When assembled, the adapter is hooked over the sample compartment.

The VGA 77 for Zeeman Adapter is shipped with the Standard Cell Holder and a bracket for the ETC 60 Workhead Mk 2.

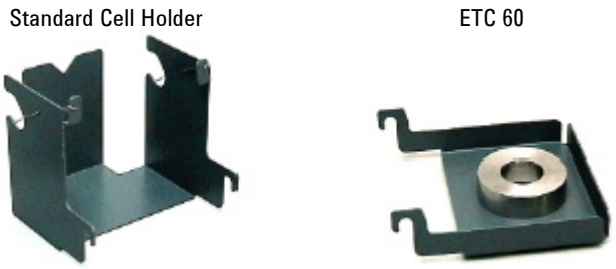


Figure 12. Standard cell holder (rear view) and ETC 60 bracket (side view)

Adapter and Standard Cell Holder Installation

NOTE It is necessary to remove the Zeeman workhead to install the adapter.

To install the adapter:

1 Remove the sampler and the Zeeman workhead from the instrument (refer to your sampler and instrument manuals for detailed instructions).

WARNING

Heavy Weight
The workhead weighs approximately 16 kg (35 lbs). To avoid injury or damage always handle carefully

NOTE The VGA 77 can be fitted to the front of the instrument as described in the instrument operation manual.

2 Make sure that the sample compartment is empty.

TIP Now is a good time to develop a method and align the lamps.

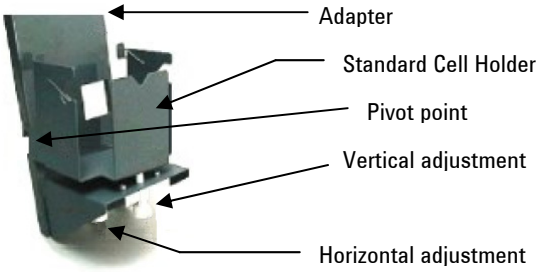


Figure 13. Adapter and standard cell holder

3 Attach the Standard Cell Holder to the adapter outside the sample compartment. Lower the hooks of the Standard Cell Holder onto the pivot points of the adapter.

4 Pass the adapter fitted with the Standard Cell Holder under the crossbar of the sample compartment.

5 Hook the adapter on the top rear edge of the sample compartment.

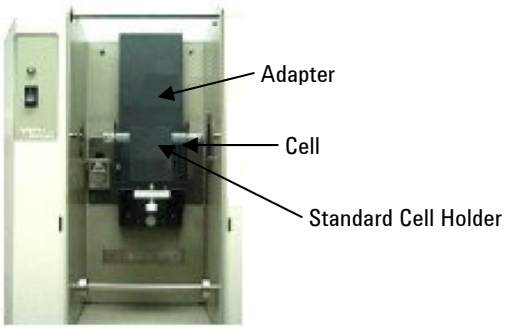


Figure 14. Adapter, standard cell holder and cell inside sample compartment

Cell Holder and Adapter Removal

The standard cell holder must be removed to allow installation of the ETC 60 Workhead Mk 2.

To remove the standard cell holder and adapter from the sample compartment:

1 Lift the cell holder up to unhook the cell holder from the adapter mounting.

2 Lift the adapter out of the sample compartment, carefully avoiding the crossbar.

Installing the Cell

This section applies to AA modules only.

NOTE While acceptable results for mercury can be obtained with the standard absorption cell, Agilent's flow-through mercury cell is recommended, since this will generally provide better analytical sensitivity and precision.
Also, for locations where the discharge of mercury vapor in nanogram quantities is not permitted, the standard absorption cell is not suitable. In these locations you should use the flow-through mercury cell together with a suitable trap (see Page 41).

WARNING

Sharp Objects
Absorption cells are fragile. Handle with care.

Installing a Standard Absorption Cell on a Mark 7 Burner

To install a standard absorption cell on a Mark 7 burner:

1. Lift the left hand spring and pass the left hand side of the cell through the cut out in the mounting bracket.
2. Gently release the left hand spring.
3. Lift the right hand spring, gently push the cell fully home and gently release the right hand spring. The cell should be positioned as shown in Figure 15.
4. Position the central inlet stem in the cell holder slot.

Figure 15. Mark 7 burner with standard absorption cell in cell holder positioned on top

Installing a Flow-through Mercury Cell on a Mark 7 Burner

To install a flow-through mercury cell (for mercury analysis) in a Mark 7 burner:

1. Open the spring clips and place the cell into position.
2. Close the spring clips.
3. Place the cell in its holder on top of the burner.

Installing a Standard Absorption Cell on Older Burners

To install a standard absorption cell on older burners (e.g., Mark VI, Mark VI-A, Mark V):

1 Lift the left hand spring clip, and pass the central inlet stem through the hole in the mounting bracket while lowering the left side of the cell into position.

2 Close the left hand spring clip.

3 Open the right hand spring clip, gently push the cell fully home and close the right hand spring clip.

4 Mount the cell in its holder on the burner.

NOTE Note that the slots on the cell holder must engage the mounting pegs on the burner clamp.

Installing a Flow-through Mercury Cell on Older Burners

To install a flow-through mercury cell (for mercury analysis) on older burners (e.g., Mark VI, Mark VI-A, Mark V):

1 Open the spring clips and place the cell into position.

2 Close the spring clips.

3 Mount the cell in its holder on the burner.

NOTE The slots on the cell holder must engage the mounting pegs on the burner clamp.

Connecting the Gas/liquid Separator Outlet

The VGA 77 is supplied with a length of black fluoro-elastomer tubing (AA only). This tubing is used to connect the vapor outlet of the gas/liquid separator to the inlet stem of the quartz absorption cell.

It is good analytical practice to keep this tubing as short as possible. Your VGA 77 should be located immediately in front of the instrument sample compartment.

AA Module

If you are using the AA reagent module, connect the outlet of the gas/liquid separator to the inlet of the cell.

To connect the outlet of the gas/liquid separator to the inlet of the cell:

1. Use a sharp scalpel to neatly cut an appropriate length of the black fluoro-elastomer tubing supplied.
2. Fit one end of the tubing over the inlet stem of the cell (for Hg cell push tubing into the inlet) and the other end over the vapor outlet of the gas/liquid separator.

NOTE When the VGA 77 is not being used, disconnect the tubing from the separator and store the cell in its holder at the front of the module.

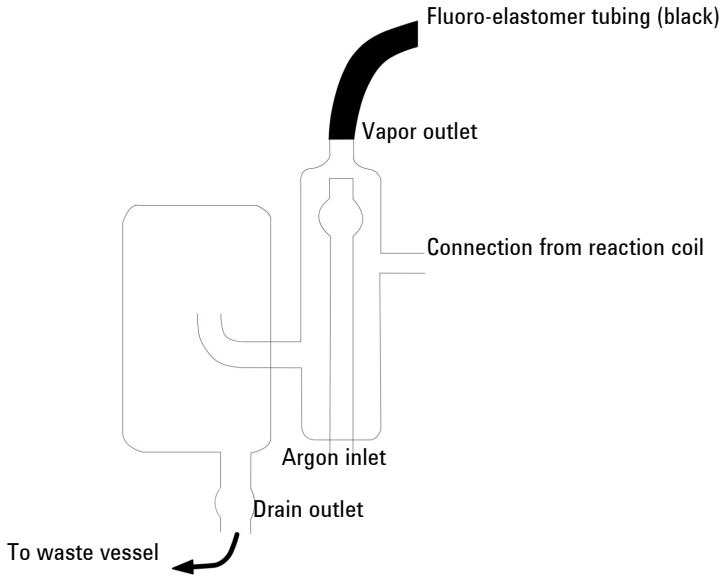


Figure 16. Gas/liquid separator for an AA module

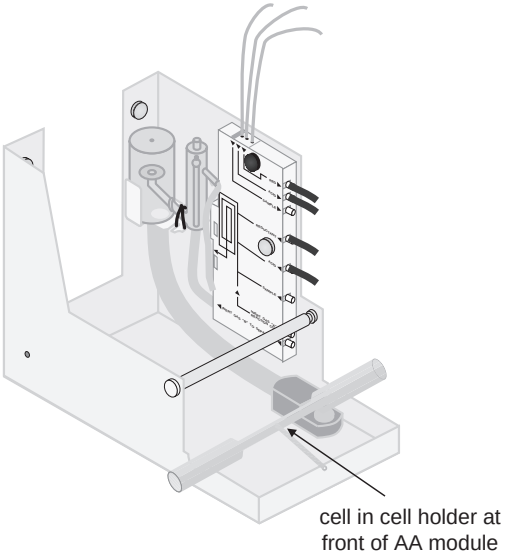


Figure 17. Cell resting in module holder when not in use

ICP Module

If you are using the ICP reagent module, connect the plasma gas/liquid separator outlet to the ICP instrument.

NOTE It is possible to connect the vapor generator to the ICP-OES systems while the plasma is on.

To connect the plasma gas/liquid separator outlet to the ICP instrument:

1. Set up the sample introduction system as described in the ICP operation manual.
2. Use a sharp scalpel to neatly cut an appropriate length of the black fluoro-elastomer tubing supplied.
3. Fit the large black fluoro-elastomer tubing over the vapor outlet of the plasma gas/liquid separator.

4 Measure the minimum length of the supplied polyethylene tubing required to connect the free end of the black fluoro-elastomer tubing to the sample inlet of the ICP nebulizer. Neatly cut the required length and make the connection.

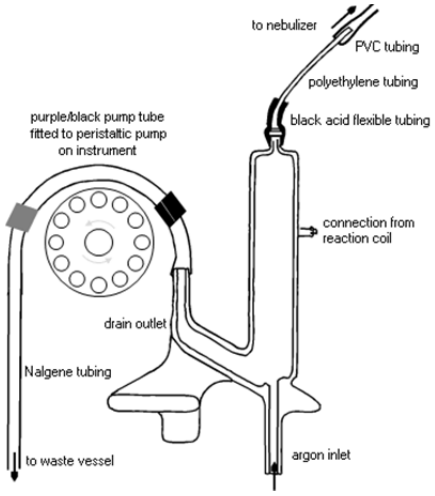


Figure 18. Plasma gas/liquid separator for ICP module

TIP For mercury analysis using $SnCl_2$, the drain peristaltic pump tubing from the spectrometer should be pinched to prevent loss of gaseous mercury through the pumped drain.

Connecting a Mercury Trap (for AA only)

Use a sharp scalpel to cut two suitable lengths of the supplied 6 mm diameter clear PVC tubing, and connect them to the two cell ports. Connect the free ends to a 'T'-piece and then to a suitable mercury trap in accordance with local regulations governing the discharge of mercury vapor.

TIP Fitting is easier if the ends of the tubing are first softened in hot water.

CAUTION Bubble type collectors must not be used, because the VGA 77 is not designed to accommodate the excessive back pressure generated by such collectors. Using bubble type collectors may lead to vapor being forced through the drain of the gas/liquid separator and results in an unreliable signal.
Collectors of loosely packed metal foil may be suitable if they comply with local laws.

Connection to the Autosampler

The VGA 77 may be connected to an Agilent or compatible autosampler for automatic sampling. Use a suitable piece of tubing to join the sample capillary on the VGA 77 to the tubing attached to the probe of the autosampler.

4. Operation

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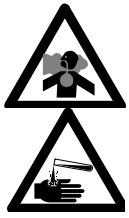
WARNING



Noxious Fumes and Corrosive Liquids

Vapor generation methods of analysis involve the generation of toxic hydrides and require the use of materials which are toxic, highly corrosive or otherwise hazardous. Careless, improper, or unskilled use of such materials can cause serious personal injury.

Always ensure that laboratory safety practices governing the use, handling and disposal of such materials are strictly observed. These safety practices should include the wearing of appropriate safety clothing.

In the VGA 77 system, highly concentrated acids are being pumped under pressure. If a leak occurs, acid could be sprayed from the system and cause serious personal injury. Always wear approved safety glasses warranted to protect the eyes.

Never pump concentrated sulfuric acid through the VGA 77 system.

In the VGA 77 system, the vapor is generated continuously while solutions are being pumped. This provides the convenience and advantages of a continuous analytical signal.

Setup

General Setup

1. Check you have the correct module installed.
2. Ensure that the inert gas supply to the VGA 77 is on, with the cylinder regulator set to the recommended pressure of 350 kPa (50 psi).
3. Ensure that the peristaltic pump is adjusted correctly as detailed in the following section.
4. Set up any other accessories you require (for example, printers or autosamplers) according to their instruction manuals.
5. Set the instrument parameters for the element to be determined. Use the integration mode with Agilent AA or ICP-OES systems. (Agilent AA users can also use PROMT mode.)
6. Fill the reagent container(s) with the required reagent(s). (Refer to Section 5.)

Pump Adjustment

To adjust the pump:

1. Set the regulator to the recommended pressure and turn on the inert gas supply.

NOTE Ensure that the inert gas supply is turned on before switching on the VGA 77 power. If you start to pump solutions through the system before the inert gas is turned on, the signal will be unreliable.

2. Select the appropriate power lead, and connect the VGA 77 to the mains power supply.
3. Place the three capillary tubes in a container of distilled water and switch the VGA 77 on. The green 'Power' indicator light is illuminated when the power is on.

NOTE The peristaltic pump will run continuously once the power is switched on.

4 Slacken both pressure adjusting screws. Swing both tube beds against the pump tubing and close the pressure bar.

5 Slowly tighten the front (sample) pressure adjusting screw until water is obviously being pumped through the tube. Tighten the pressure adjusting screw a further half turn. At this setting the pumping rate will be close to optimum. Any further tightening of the screw will tend to reduce the pumping rate.

CAUTION Do not over-tighten the pressure adjusting screws. Excessive tightening will shorten the life of the pump tubes and could cause permanent damage to the pump.

NOTE At this stage, inert gas may be seen bubbling out of both the other tubes. This is a normal consequence of pressurizing the system and this situation is automatically corrected when you adjust the pumping pressure in these tubes.

TIP To see if the liquid is being pumped smoothly, you can deliberately create air bubbles by dipping the capillary in and out of the container.

6 Repeat Step 5 with the rear pressure adjusting screw.

7 Measure the uptake rate through the sample pump tubes. The uptake rate should be within the range 6–8 mL/min.

NOTE You can do this by filling a suitable measuring cylinder to a convenient reference mark with distilled water, then placing the sample capillary into the cylinder and observing the volume of water pumped out of the cylinder over a timed interval.

With a 50 Hz power supply the uptake rate should be about 7 mL per minute; with a 60 Hz power supply it should be about 8 mL per minute.

NOTE Note the exact flow rates for future reference in the AA performance log in Appendix A at the end of this manual.

8 Similarly measure the uptake rate for each of the other pump tubes. The uptake rate for each should be within the range 0.8–1.2 mL/min.

NOTE Note the exact flow rates for future reference in the AA performance log in Appendix A at the end of this manual.

CAUTION If you are using the VGA 77 with an ICP system, you must now adjust the speed of the instrument peristaltic pump until liquid is being pumped out of the gas/liquid separator faster than it is being pumped in by the VGA 77. Set the spectrometer Pump speed to a minimum of 35 rpm.

Your VGA 77 is now ready for use. You should now either:

- Proceed to 'AA Setup' on the following page, if you are using the VGA 77 with an AA system
- Proceed to 'ICP setup on Page 49, if you are using the VGA 77 with an ICP system, or
- Carry out the instructions for closing down the VGA 77 on Page 53.

AA Setup

1 Remove the cell in its holder from the burner and place it at the front of the reagent module.

2 Lower the burner so that it is clear of the optical path.

NOTE Agilent recommends using reduced slit height (0.5) in all methods.

3 Fit the required hollow cathode lamp and set up the spectrometer as described in your instrument operation manual.

4 Select **Optimize > Optimize Lamp**.

5 Position the cell in its holder on top of the burner.

6 Align the cell in the optical path as follows:

   a Hold a piece of white card between the right-hand end of the absorption cell and the sample compartment window.

   b Using the burner positioning controls, adjust the position of the cell until the light from the hollow cathode lamp passes through the cell onto the card. Remove the card.

   c Use the burner positioning controls to adjust the position of the cell for maximum transmission.

For mercury determinations, proceed to ‘Conditioning the System’ on Page 48.

Lighting the Flame

To light the flame:

1 Carry out any safety checks described in your spectrometer operation manual. Follow all instructions and observe all warnings.

2 Set the acetylene flow rather higher than normal in order to obtain a fuel-rich air-acetylene flame. This is required for igniting the flame.

NOTE Some instruments may automatically set a fuel-rich mixture. You should refer to your spectrometer operation manual for details.

WARNING

Explosion Hazard
With the VGA 77 cell installed, the burner will be lower than usual, and a standard mixture or a fuel-lean mixture may not ignite readily. To avoid an explosive accumulation of fuel and oxidant in the sample compartment, ALWAYS ensure that the gas mixture is fuel-rich before operating the Ignite button.

3 If a Mark VI burner is fitted, tilt the absorption cell clear of the burner. (On older instruments this will also ensure that the igniter arm does not strike the cell.)

CAUTION If you leave the absorption cell in a fuel-rich flame, carbon particles may be deposited on the outside wall of the cell. Under these conditions, the cell can be permanently damaged by local overheating.

4 Ignite the flame and adjust the fuel flow to give a lean flame. If necessary, lower the cell back to its normal position.

Conditioning the System

The objective of this procedure is to 'condition' the complete system and obtain a stable signal before calibrating the instrument and analyzing your samples.

This procedure should be carried out:

- When the system is first installed
- After the system has been completely flushed and cleaned

- When a new absorption cell (or flow-through mercury cell) is fitted
- Whenever new pump tubes are fitted
- When the reaction coil is renewed

To condition the system:

1. Allow the pump to operate for three or four minutes to stabilize the flow rates.
2. Make sure the instrument is in 'Abs' mode with a suitably short integration period.
3. Aspirate your highest standard, monitor the signal, and measure the time taken for the signal to rise from zero to its stable value. The time you measure for this interval will be the pre-read delay that you should enter into your system software.

NOTE When presenting solutions to the system, always wait until this period has elapsed before taking a reading.

4. Aspirate your analytical blank
5. Continue to aspirate the standard and analytical blank in turn until a consistent response is obtained for the standard solution and then proceed to 'Operation' on Page 51.

If a consistent response cannot be obtained within a reasonable time, contamination is the likely cause. Even slight contamination of the system can suppress production of hydride (or mercury vapor). In this circumstance, you must suspend the conditioning procedure and clean the system thoroughly before continuing. Refer to Page 71 for cleaning instructions.

ICP Setup

The following procedure conditions the ICP system for the vapor generation analysis. This procedure should be carried out:

- When the system is first installed
- After the system has been completely flushed and cleaned

- When new pump tubes are fitted
- When the reaction coil is renewed
- Before each analysis

To set up the ICP:

1 Ensure that the plasma is on and the peristaltic pump is operating.

2 Allow the pump to operate for three or four minutes to stabilize the flow rates.

3 Perform a time scan on the Time Display page in ICP Expert II and on the Conditions page in ICP Expert. See the online Help for more information. View the signal as you alternate between the blank and top calibration standard. Use the cursor to determine the suitable delay time required.

NOTE Due to the high sensitivity of the VGA 77/ICP-OES system, the upper concentration of standards used for calibration is limited (by memory effects etc.). For hydride forming elements, use a top standard of about 100 ppb.

4 Continue the previous step until a consistent response is obtained. When the response is consistent, the system is ready for use. Remember to optimize the operating conditions for your program. Pay particular attention to sample delay, stabilization and rinse times.

If a consistent response cannot be obtained within a reasonable time, contamination is the likely cause. Even slight contamination of the system can suppress production of hydride (or mercury vapor). If this happens, stop the conditioning procedure and clean the system thoroughly before continuing. Refer to Page 71 for VGA cleaning instructions, and refer to your instrument manual for details on cleaning your ICP.

Operation

Before starting the analytical program you must measure the analytical signal caused by the presence of analyte in:

- The acid and reductant solutions being pumped through the system
- The analytical blank solution

To do this:

1. Place all three uptake tubes in a single container of distilled water. Wait for about 40 seconds to allow the system to stabilize. Perform an instrument zero.
2. Leave the sample uptake tube in distilled water and place the acid and reductant uptake tubes in their respective reagent solutions. Wait for about 40 seconds and measure the signal. Any signal registered is attributable to analyte in the solutions being pumped through the system.
3. Place the sample uptake tube in the analytical blank solution. Wait for about 40 seconds and measure the signal. This measurement is the total signal attributable to analyte in all solutions used (acid, reductant, analytical blank).
4. The measurements obtained in the previous two steps allow you to decide whether the blank signal is acceptable or not before using the blank solution to calibrate the instrument at analytical zero.

You can now calibrate the system and measure your samples.

Samples can be presented to the system manually by dipping the sample capillary tube into a flask, test tube or vial. You can use the VGA 77 with non-Agilent instruments and autosamplers.

The general procedure for using automatic samplers is given in the next section.

Automatic Operation

CAUTION Operation of the VGA 77 is not interlocked to the system. In the event of gas supply failure, pumping faults, or if the reagent containers are emptied, the program will not be automatically stopped and results from the program will not be analytically valid.

CAUTION For ICP systems. At the end of an automatic run, the VGA 77 pump continues to run and gas continues to flow. To avoid waste liquid flooding the system, set the 'Pump rate at end of sequence' on the Preferences page in ICP Expert II or on the Sequence page in ICP Expert to a rate that ensures the removal of all waste liquid from the gas/liquid separator (typically 40–45 rpm).

Observe the following general rules, but refer to the appropriate instrument operation or system manual for specific operating instructions.

- The VGA 77 technique requires the use of acids at high concentrations. You must therefore ensure that the sample probe on the autosampler is corrosion-resistant. The Agilent SPS 3 is fitted with a corrosion-resistant probe. This probe should always be used for VGA 77 applications.
- When connecting the autosampler to the VGA 77 uptake capillary, ensure that the joint is leak-free and offers minimal dead volume by keeping the connecting tube as short as possible. The uptake from the autosampler to the vapor generator is the only connection required between VGA 77 and other system components—no electrical interconnection is required.
- Remember to enter a suitable delay time to allow for the extra time needed for the solution to emerge from the autosampler tubing. Also check the stabilization times.
- In the software, select the required number of replicate measurements to be taken and select the appropriate re-calibration and reslope frequencies.

- Load the autosampler with your analytical blank, standard solutions and samples in the correct locations.
- Save your revised method.
- Before starting the analytical program, carefully check all sampling parameters to ensure that they are correct. Also check that the reagent containers are full.

Shutting Down

1. Turn off the flame/torch as described in your instrument operation manual. Leave the VGA 77 switched on. You will also need to leave the ICP pump running.
2. Run a concentrated hydrochloric acid solution through the system for 2 to 5 minutes then immerse the three uptake capillary tubes in a container of distilled water (at least 200 mL) and leave the pump running for 10 to 20 minutes to flush the system.
3. Remove the capillary tubes from the distilled water. Leave the pump running until the distilled water has been pumped through the system.
4. Switch the VGA 77 off, but leave the inert gas supply on at the regulator. Open the pressure bar and swing the pump tube beds clear of the pump rollers. Remove the pump tubes from the pump rollers and release them from the retaining bracket. Ensure that they are kept completely clear of the rollers while you carry out the next step.
5. Switch the VGA 77 on. This will allow inert gas to flow through the entire system and thus minimize the possibility of residual solutions settling in the lowest section of the tubing. Allow the inert gas to flow for about five minutes.
6. Switch off the VGA 77, and the ICP pump, if necessary.
7. Turn off the inert gas supply at the regulator.

NOTE Inert gas will flow through the separator even with the VGA 77 switched off.

8 Clean all components as described on Page 71.

9 Clean your instrument according to its operation manual.

WARNING

Hot surface

For AA systems. Be careful when touching the absorption cell or the burner, as they may be very hot. Contact with these components can cause severe burns. Always ensure that these components are allowed to cool before attempting to remove them from the sample compartment.

Changing Modules

The modular design of the VGA 77 allows easy changing of modules.

To change modules:

1 Shut down the system as described in the previous section.

2 Disconnect the sample inlet to gas/liquid separator tubing from the sample inlet, using the reverse of the connection procedure on Page 38.

3 If you are removing an ICP module, remove the drain tubing from the instrument pump.

4 Disconnect the inert gas connections from the pump unit to the module (refer to Page 24, Steps 4 and 5).

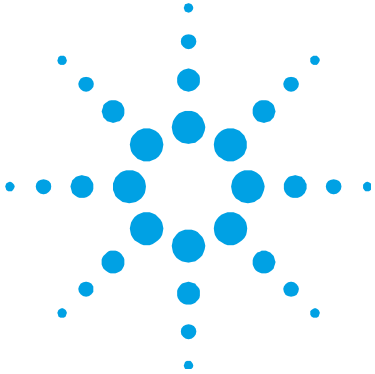
5 Undo the locking screw on the bottom left of the VGA 77 frame.

6 Ensure the drain tube is completely empty, and remove it from the module.

7 Lift the module up to release the pins at the back of the module from the VGA 77 frame, then remove the module.

8 Immerse the removed module in distilled water, and back flush the lines to clear them of acid remnants. Dry the module in air before using again.

9 Install the required module, following the procedures described in Section 3.



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Background

The VGA 77 employs continuous flow technology where samples and liquid reagents are pumped together and mixed. The gaseous reactions products are swept by a flow of argon gas into the spectrometer.

NOTE Nitrogen gas can be used with the AA reagent module.

The following figure shows a schematic diagram of the VGA 77.

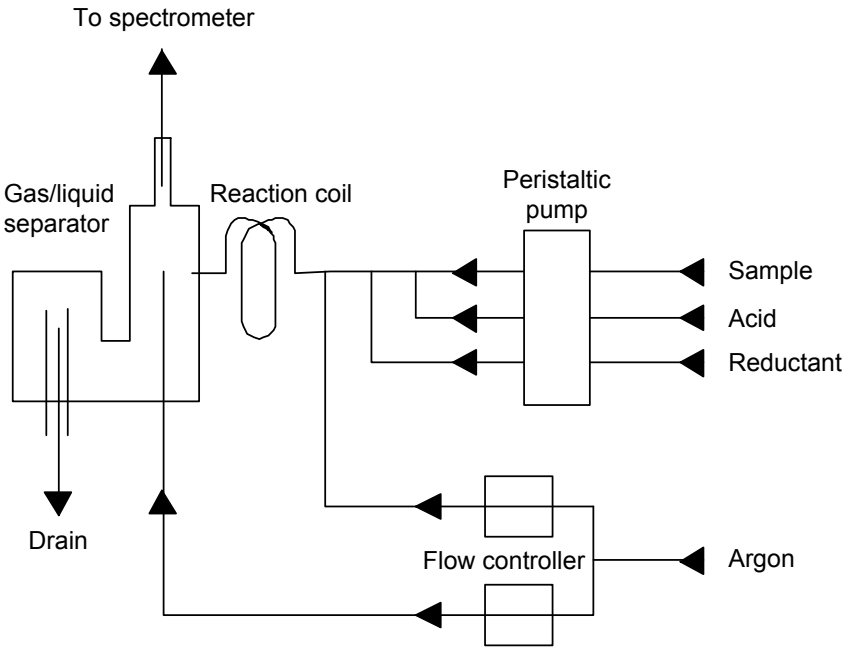

Figure 19. Schematic of the VGA 77

The peristaltic pump maintains a constant flow of analytical solutions into the accessory.

The sample and acid are allowed to merge first before the reductant, usually sodium borohydride ($NaBH_4$), enters the stream. (For mercury analysis, $SnCl_2$ solution is recommended.) Argon is then introduced into the liquid stream and the reaction proceeds while the mixture is flowing through the reaction coil. Vigorous evolution of hydrogen assists the stripping of the hydride (or mercury vapor) from the liquid into the argon.

The gas is then separated from the liquid in the separator. The liquid drains away to waste. At this point a second stream of argon is introduced to ensure that the gas stream is not saturated with water vapor and does not condense in the sample introduction system.

The gas containing the element of interest then passes out of the separator into the spectrometer where it is analyzed.

As the VGA 77 produces a continuous signal, you should use the integration mode with Agilent AA or ICP-OES systems. (Agilent AA users can also use PROMT mode.)

Factors Affecting the Formation of Hydrides

Acid Concentration

The concentration of acid will affect the efficiency of hydride formation in the VGA 77. You must ensure that the acid concentrations of blank, standard and sample solutions are the same.

For multi-element analysis, a compromise acid concentration will be required.

NOTE The use of oxidizing acids (e.g. H_2SO_4, $HClO_4$) should be avoided.

Oxidation State

The hydride-forming elements may exist in more than one oxidation state in samples and standards. This can have dramatic effects on the measured signal.

For example, for Te and Se, a 10x or more increase in the signal intensity is observed if the lower oxidation state is prepared.

Solutions containing H_2SO_4 and $HCLO_4$ acids may give little or no signal for any hydride-forming elements. The use of H_2SO_4 and $HClO_4$ is to be avoided as these are very strong oxidizing agents.

Sample and standard and blank solutions must be prepared by a similar method to obtain the lower oxidation state.

Interferences

The measurement of some elements by VGA can be affected by the presence of other elements or molecules in the sample matrix. These reduce the amount of element detected by the spectrometer by adversely affecting the amount of hydride formed.

A list of observed interferences is given in the following table.

Table 1. % Loss of analyte signal due to element interferences

Analyte	>50%	10–50%	10%
As	Au, Ge, Ni, Pt, Pd, Rh, Ru	Ag, Bi, Co, Cu, Sb, Se, Sn, Te	Al, B, Ba, Be, Ca, Cd, Cr, Cs, Fe, Ga, Hf, Hg, In, Ir, K, La, Li, Mg, Mn, Mo, Na, Pb, Rb, Re, Si, Sr, Ti, Tl, V, W, Y, Zn, Zr
Bi	Ag, Au, Co, Cu, Ni, Pd, Pt, Rh, Ru, Se, Te	As, Cd, Cr, Fe, Ge, Ir, Mo, Sb, Sn	Al, B, Ba, Be, Ca, Cs, Ga, Hf, Hg, In, K, La, Li, Mg, Mn, Na, Pb, Rb, Re, Si, Sr, Ti, Tl, V, W, Y, Zn, Zr
Ge	As, Au, Cd, Co, Fe, Ni, Pd, Pt, Rh, Ru, Sb, Se, Sn	Bi, Cu, Ir, Te	Ag, Al, B, Ba, Be, Ca, Cr, Cs, Ga, Hf, Hg, In, K, La, Li, Mg, Mn, Mo, Na, Pb, Rb, Re, Si, Sr, Ti, Tl, V, W, Y, Zn, Zr
Sb	Au, Co, Ge, Ni, Pt, Pd, Rh, Ru	As, As, Cr, Cu, Re, Se, Sn	Al, B, Ba, Bi, Ca, Cd, Cs, Fe, Be, Ga, Hf, Hg, In, Ir, K, La, Li, Mg, Mn, Mo, Na, Pb, Rb, Si, Sr, Te, Ti, Tl, V, W, Y, Zn, Zr
Se	Ag, Cu, Ni, Pd, Pt, Rh, Ru, Sn	Au, As, Cd, Co, Fe, Ge, Pb, Sb, Zn	Al, B, Ba, Be, Bi, Ca, Cr, Cs, Ga, Hf, Hg, In, Ir, K, La, Li, Mg, Mn, Mo, Na, Rb, Re, Si, Sr, (Te), Ti, Tl, V, W, Y, Zr
Te	Ag, Au, Cd, Co, Cu, Fe, Ge, In, Ni, Pb, Pd, Pt, Re, Rh, Ru, Se, Sn	As, Bi, Ir, Mo, Sb, Si, W	Al, B, Ba, Be, Ca, Cr, Cs, Ga, Hf, Hg, K, La, Li, Mg, Mn, Na, Rb, Sr, Ti, V, Y, Zn, Zr

Contamination

When first establishing your analytical method you must always check for contamination before carrying out your program.

NOTE Traces of potassium iodide (KI) will interfere severely with determinations for bismuth, mercury, selenium and tellurium. You must always ensure that the VGA 77 system is completely free of residual iodide ions (e.g., KI) before attempting to determine these elements.

The most practical way of minimizing contamination problems is to provide a separate module (including the reagent containers and pump tubes) for these elements. Page 79 lists spares and part numbers.

Another form of contamination can occur when changing from a high level standard to a low level standard. You must make sure that all tubing in the module is thoroughly rinsed.

Memory Effects

Because the VGA 77 uses a chemical reaction to produce the elemental hydrides, some memory effects can be encountered in the reaction products when changing between low and high level standards. Always allow several minutes when moving from a high to a low level standard.

You should also ensure that the standards are prepared at the same concentration as the samples.

Certain types of plastic can cause memory effects for mercury. Keep mercury top standard concentration as low as practicable.

Background Absorption (AA only)

In general, background correction is not necessary for vapor generation work. Occasional exceptions may be encountered. For example, when determining low level arsenic in the presence of another hydride forming element.

You should establish whether background absorption is occurring before carrying out your analytical program. This can be done by analyzing a sample with background correction on and then repeating the analysis with it off. (Remember to do an instrument zero before each measurement.)

If a significant difference is found between the two measurements, then perform your analyses with background correction on. If there is none, leave the background correction off.

Laboratory Procedures

General

The vapor generation technique demands a particularly high standard of care in all of the activities which affect the accuracy and precision of the analytical result. Scrupulous cleanliness is essential in all laboratory procedures; standards and samples must be meticulously prepared and carefully handled. Strict precautions must be taken to avoid contamination of apparatus and even though laboratory ware is stored under ideal conditions, it should be thoroughly re-washed before use.

Strict care should also be taken to avoid contamination of all reagents and distilled water. Ideally, reagents should be entirely free of the element of interest, but this is obviously impossible for all analyte elements in all reagents. Consequently, you must always establish the level of the analytical signal attributable to analyte in the reagents. It is, of course, standard practice to check the analytical signal from the blank solution before calibrating the instrument and carrying out the analytical program.

Dispose of waste in accordance with relevant safety practices.

Standards

Prepare your calibration standards from stock solutions.

For some samples it will be necessary to compare the calibration slopes using the normal calibration method with those obtained using the standard additions method. If the slope is not the same, you should use the standard additions technique for the analysis.

Reductant—Sodium Borohydride

The recommended sodium borohydride ($NaBH_4$) concentration is 0.6% w/v. However better results will be obtained for difficult samples containing high concentrations of metals if the sodium borohydride concentration is reduced to 0.3% w/v.

NOTE Stannous chloride reductant is recommended for mercury determinations. See Page 64.

Always stabilize the solution by first adding sodium hydroxide (NaOH 0.5% w/v). Since sodium borohydride will decompose significantly in one or two days, you should not prepare more than 500 mL at a time. At a flow rate of about 1 mL per minute, this should be enough for continuous operation over a typical working day. Stability may be improved by passing the solution through a 5 micron filter. You can also extend the working life of the solution by storing at 5 °C. (The solution should remain stable for approximately one week if stored at 5 °C.) Allow the solution to reach room temperature prior to analysis.

Pump Tubing

The pump tubing should be checked regularly by checking the flow-rate (see Page 44 ‘Pump adjustment’).

NOTE When concentrated acid is first pumped through the acid tube, the inside of the tubing may turn white, but this does not impair the efficiency of the tube.

Ensure samples, reagents and standards are at room temperature prior to analysis. The VGA 77 pump will not operate correctly with hot or cold solutions as the pumping rate may vary with solution temperature.

Sources (AA only)

Hollow cathode lamps or UltrAA lamps can be used for all VGA 77 methods for AA analysis.

Basic Methods

The hydride-forming elements may exist in more than one oxidation state in samples and standards. (See Page 57.) The following analytical methods for sample and standard have been developed to ensure samples and standards are present in the correct oxidation state.

Antimony

Prepare samples in at least 1 M hydrochloric acid and ensure that any analyte present as Sb^{V} is reduced to Sb^{III} by the action of potassium iodide at a concentration of 1% w/v. Reduction is spontaneous and heating should not be required.

Reductant container:	$NaBH_4$ 0.6% w/v, NaOH 0.5% w/v
Acid container:	5 to 10 M HCl

Arsenic

NOTE If arsenic and selenium are both to be determined from the same sample, determine selenium first, and avoid KI in samples or standards. You can then determine arsenic after the KI reduction step and any other appropriate treatment (such as the addition of urea if excess nitric acid is present).

Arsenic in the sample must be in the inorganic form, otherwise digestion will be necessary.

If digestion is necessary, use acid digestion, ensuring no residual oxidizing acid is present (see 'Oxidation States' on Page 57, or ashing with an appropriate ashing aid. Simple dry ashing is not recommended.

Prepare samples in at least 1 M hydrochloric acid.

Ensure that any analyte present as As^{V} is reduced to As^{III} by the action of potassium iodide at a concentration of 1% w/v.

Reduction will take about 50 minutes at room temperature. The reduction can also be carried out at 70 °C in about four minutes; however you must cool the samples and standards to room temperature prior to analysis. Since the pumping rate may vary with solution temperature, the VGA 77 pump will not operate correctly with hot solutions.

If the reduction step is omitted, and the analyte is retained as As^{V}, the analytical sensitivity is about 20-30% of that obtained for As^{III}. If the original solution contains As^{III}, reduction by potassium iodide is not required.

Reductant container:	$NaBH_4$ 0.6% w/v (see also above) NaOH 0.5% w/v
Acid container:	5 to 10 M HCl

Bismuth

The presence of potassium iodide will severely suppress the analytical response. Always ensure that the VGA 77 system is completely free of potassium iodide before performing bismuth determinations.

NOTE To avoid contamination, it is recommended that you dedicate a separate module to the analysis of those elements requiring pre-reduction with KI—As and Sb.

Prepare samples in 1 M hydrochloric acid.

Reductant container:	$NaBH_4$ 0.6% w/v NaOH 0.5% w/v
Acid container:	5 M HCl (Higher acid concentrations will depress the analytical signal.)

Mercury

Traces of potassium iodide will interfere severely with the production of mercury vapor and the analytical response may be completely suppressed. Always ensure that the VGA 77 system is completely free of potassium iodide before performing mercury determinations.

NOTE To avoid contamination, it is recommended that you dedicate a separate module to the analysis of those elements requiring pre-reduction with KI—As and Sb.

The mercury in the sample must be in the inorganic form, otherwise digestion or use of releasing agents (e.g. $CdCl_2$), will be necessary.

If digestion is necessary, use acid digestion, or ashing with an appropriate ashing aid. Simple dry ashing is not recommended as the mercury will be lost.

Dilute mercury solutions tend to be unstable; all analytical solutions should be stabilized by the addition of nitric acid (5% v/v) and hydrochloric acid (5% v/v). Prepare fresh standards daily.

If you are using the AA module:

Acceptable results can be obtained with the standard absorption cell, however the flow-through mercury cell is recommended since this will generally provide better analytical sensitivity and precision.

AA mercury determination is a cold vapor technique. Mercury is best determined with a cold cell as the analytical sensitivity is reduced when the cell is heated. Under no circumstances should the flow-through cell be heated.

CAUTION Heating the flow-through cell may damage it.

If you are experiencing problems with carryover of water vapor into the cell (or condensation in the cell), heating of the flow cell can eliminate this. One approach to heating the cell is to use the ETC 60 Electrothermal Temperature Controller programmed at 300 °C. Alternately, another switch in heat source (like a lamp) can reduce the occurrence of this problem.

For locations where the discharge of mercury vapor in nanogram quantities is not permitted, the standard absorption cell is not suitable and you should use the flow-through mercury cell together with a suitable trap. Refer to Page 41.

Mercury can be determined using stannous chloride as the reductant.

Reductant container:	$SnCl_2$ (25% w/v) in HCl (20% v/v)
Acid container:	H_2O

NOTE To prepare the $SnCl_2$ solution, add $SnCl_2$ crystals to concentrated HCl and warm the mixture on a hot plate to complete dissolution before adding the water. A piece of granulated tin added to the mixture will reduce any Sn^{IV} to Sn^{II} and produce a clear solution

NOTE Mercury suffers from memory effects as it adheres to plastic. Keep concentrations low to minimize these memory effects.

An alternative method for mercury determination with AA uses the $NaBH_4$ reduction procedure. In this method, it is preferable to use a lower concentration of sodium borohydride than would normally be used for the hydride-forming elements. The concentration of acid pumped through the VGA 77 system is generally not critical.

For ICP analysis, ensure the spray chamber drain is clamped to avoid loss of the mercury analyte.

Reductant container:	$NaBH_4$ 0.3% w/v NaOH 0.5% w/v
Acid container:	5 M HCl

A lower analytical signal may be obtained with this method than with the stannous chloride method.

Selenium

NOTE If arsenic and selenium are both to be determined see Page 69 'Additional Notes' at the end of this section.

The selenium in the sample must be in the inorganic form, otherwise digestion will be necessary.

If digestion is necessary, use acid digestion, or ashing with an appropriate ashing aid. Simple dry ashing is not recommended because selenium is highly volatile and recovery will be poor.

Se^{VI} is not quantitatively recovered by hydride generation and must be reduced to Se^{IV} by warming with concentrated hydrochloric acid. Prepare the samples in 6-7 M hydrochloric acid (use of a lower acid concentration will result in greater inter-element interferences), heat at 70-90 °C for at least 10 minutes. Cool to room temperature before analysis.

Reductant container:	$NaBH_4$ 0.6% w/v NaOH 0.5% w/v
Acid container:	10 M HCl

Tellurium

The presence of potassium iodide will severely suppress the analytical response. Always ensure that the VGA 77 system is completely free of potassium iodide before performing tellurium determinations.

NOTE To avoid contamination, it is recommended that you dedicate a separate module to the analysis of those elements requiring pre-reduction with KI (ie., As or Sb).

Te^{VI} is not quantitatively recovered by hydride generation and must be reduced to Te^{IV}. Prepare the samples in 6-7 M hydrochloric acid, heat at 70-90 °C for at least 10 minutes and cool to room temperature before analysis.

Reductant container:	$NaBH_4$ 0.6% w/b NaOH 0.5% w/v
Acid container:	5 M HCl (Higher acid concentrations will reduce the analytical signal.)

Tin

The best results for tin will generally be obtained from solutions prepared in 1% w/v tartaric acid. Also, a study[(2)] has shown that the addition of L-cysteine greatly improves tin determination by hydride generation. The L-cysteine reduces metal interferences and improves precision and sensitivity. Some improvement in calibration linearity has also been noted.

The concentration of acid pumped into the VGA 77 is also critical in tin determinations, and the analytical signal will be severely depressed at concentrations higher than 0.5 M. (See Page 57 'Acid Concentration'.)

When determining tin using the AA technique, it is essential to determine tin at the 286.3 nm wavelength and 0.2 nm slit since this will provide the best combination of analytical sensitivity and dynamic range. The 235.5 nm line commonly used for flame and furnace AA gives very poor sensitivity and the 224.6 nm line shows very little increase in analytical response above approximately 0.3 absorbance.

Reductant container:	$NaBH_4$ 0.6% w/v NaOH 0.5% w/v
Acid container:	0.5 M HCl
Sample	1% Tartaric acid controlled pH (2.0 to 3.0)

Alternative method[2]

Reductant container:	$NaBH_4$ 0.5% w/v NaOH 0.1% w/v
Acid container:	D.I. H_2O
Sample	1% w/v Tartaric acid 1% w/v L-cysteine

Additional Notes

- If arsenic and selenium are both to be determined from the same sample, determine selenium first and avoid KI in samples or standards. You can then determine arsenic after the KI reduction step and any other appropriate treatment (such as the addition of urea if excess nitric acid is present).
- For the determination of arsenic and selenium in many practical samples containing high concentrations of metals such as copper, iron or nickel, fewer interferences have been observed using 0.3% w/v $NaBH_4$ solution concentration (rather than 0.6% w/v). Use of the lower reductant concentration may be less sensitive.
- You can minimize interferences of transition metals such as cobalt, copper, iron, and nickel on arsenic and selenium by preparing samples in 6-7 M HCl. With lower acid concentrations, greater inter-element interferences are present.
- Co-precipitation methods using lanthanum compounds have also been found to be useful.

References

1 Smith, A.E., 'Interferences in the determination of elements that form volatile hydrides with $NaBH_4$ using AAS and Ar/H_2 flame.', *Analyst*, May, **1975**, 100, 300-306

2 Determination of tin by hydride generation AA, **1992**, October, No. 107

Additional References

The following references are recommended:

- *Spectroscopy*, **1985**, Vol 1 (0), 60
- Appl. Spectrosc., **1985**, Vol 39 (1), 48
- Jiri Dedina and Dimiter L. Tsalev, Hydride Generation Atomic Absorption Spectrometry. Published by John Wiley and Sons, Edited by J.D. Winefordner. ISDN 0 471 953644

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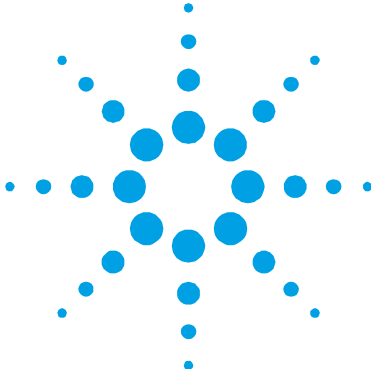

6. Maintenance and Spare Parts

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Cleaning Procedures

General

The VGA 77 uses high concentrations of HCl. Continual exposure to HCl fumes may damage the instrumentation. Maintain an efficient exhaust system during VGA determinations and remove HCl solutions from the VGA 77 and the spectrometer when you finish your analyses.

Always clean up any spilt liquids immediately. The front of the spectrometer, the burner/torch compartment and the VGA 77 should be cleaned daily with a soft cloth. If necessary, use a cloth dampened with distilled water.

Follow the shut down procedure as described on Page 53.

Absorption Cell

This section applies to AA modules only.

Always clean the absorption cell thoroughly immediately after use, when contamination is suspected while conditioning the system, or whenever the system has been left unused for some time. Throughout the cleaning procedure remember that the cell is fragile and must be handled with appropriate care.

WARNING

Hot surface

Before touching the cell remember that it may be very hot and could cause serious burns. Always turn the flame off and allow the cell to cool before attempting to remove it from the sample compartment.

1. Disconnect the black fluoro-elastomer tubing from the cell inlet.
2. Release the spring clips and remove the absorption cell from the cell support.
3. Soak the absorption cell in dilute nitric acid for at least 30 minutes.
4. Rinse thoroughly with distilled water and allow it to air dry in a dust-free location.
5. If the cell has been exposed to contamination with potassium iodide, soak the cell in sodium hydroxide solution (0.5% w/v) for at least 30 minutes. Wash thoroughly with dilute hydrochloric acid and then wash thoroughly with distilled water. Allow to air dry in a dust-free location.

CAUTION Do not leave the cell in the sodium hydroxide for too long because this solution may accelerate cell devitrification. Avoid handling the central part of the quartz cell with bare hands after it has been cleaned or while it is in use.

The working life of the cell will depend on the type of analytical programs carried out. At some stage, the glass will start to turn white indicating the onset of devitrification. Initially, devitrification will not degrade the analytical performance, but eventually the devitrified area will extend over most of the cell at which point it may be necessary to discard the cell.

If devitrification is excessive, the cell will eventually crack spontaneously or fail through thermal shock.

Whether the cell becomes devitrified or not, and even with meticulous cleaning, the inside surface may deteriorate to the extent that the analytical signal will be suppressed. You should regularly monitor the performance of the cell using absorbance measurements obtained from standard solutions. Discard the cell as soon as the results of this monitoring become unacceptable.

NOTE When a significant number of samples are being analyzed, the use of a Drierite trap inserted between the absorption cell and the output of the gas/liquid separator will protect the absorption cell and extend its lifetime. (Refer to 'Use of Drierite Trap to Extend the Lifetime of Vapor Generation Absorption Cell', 1986, October, No. 65 on www.agilent.com.)

Under some circumstances, the center section of the cell may gradually soften in the flame and sag. If uncorrected, this may become so severe that alignment of the cell within the optical path is impossible. When sagging is first noticed, it can be corrected by simply turning the cell over so that it will return to its normal shape the next time it is heated.

Gas/Liquid Separator

As with the cell, the gas/liquid separator must be cleaned immediately if contamination is suspected during conditioning, or whenever the system has been left unused for some time. Do not allow hydrochloric acid solutions to remain in the separator.

WARNING

Corrosive Chemicals

Contact with concentrated acids can cause severe chemical burns. Always wear protective clothing and gloves while cleaning the separator.

To clean the gas/liquid separator:

1. Disconnect the tubing from the vapor outlet of the separator.
2. Disconnect the tubing from the sample inlet of the separator.
3. Carefully remove the rubber mounting strap holding the separator in place.
4. Disconnect the drain tubing from the drain in the bottom of the module. You may now lift the separator out of the module.
5. Disconnect the tubing from the inert gas inlet of the separator.
6. If the separator has been exposed to solutions containing potassium iodide, soak the separator in sodium hydroxide solution (0.5% w/v) for at least 30 minutes. Wash thoroughly with dilute hydrochloric acid and then wash thoroughly with distilled water. Allow to air dry in a dust-free location.

NOTE If the separator has not been exposed to potassium iodide solutions, it may be treated in the same way as the absorption cell. To avoid KI contamination, it is recommended that you dedicate a separate module to the analysis of those elements requiring pre-reduction with KI (i.e., As or Sb).

7. Refit the separator into its mounting bracket, secure it with the retaining strap and reconnect all tubing. If the system is not to be used for some time, the separator should be filled with distilled water.

Tubing

Pump Tubes

When the system is not being used, the pressure bar should be released, the pump tubes removed from around the rollers and released from the retaining bracket. This will minimize distortion of the tubes and help to prolong their working life.

To reduce mechanical wear of the pump tubes, the outside of the tubes and the surface of the pump rollers should be sprayed daily with a silicone lubricant.

If the procedure specified in 'Shutting Down' on Page 53 is followed, the system tubing will generally remain clean, however the efficiency of all pump tubes will eventually be degraded to the point at which they must be replaced. You should regularly monitor the performance of each pump tube using flow rate measurements (see Page 44). Discard the pump tubes as soon as the results of this monitoring become unacceptable.

NOTE The tubes connecting the black-black pump tubes to the pump module may occasionally need to be replaced. To do this, cut four 2 cm lengths of the thinner black fluoro-elastomer tubing supplied. Place these over each end of two of the black-black pump tubes.

Fluoro-elastomer Tubing

The fluoro-elastomer tubing connecting the gas/liquid separator to the absorption cell is particularly susceptible to contamination with potassium iodide. To avoid this, it is recommended that you dedicate a separate module to the analysis of those elements requiring pre-reduction with KI (i.e., As or Sb).

If there is no KI contamination, you can clean the tubing by flushing it well with distilled water.

If there is KI contamination, you need to remove traces of KI as follows:

1 Disconnect and remove the tubing.

2 Soak the tubes in sodium hydroxide solution (0.5% w/v). Wash thoroughly with dilute hydrochloric acid and then wash thoroughly with distilled water.

3 Allow the tubes to air dry in a dust-free location.

4 Reconnect the tubing.

An alternative method of removing traces of iodine from the tubing and gas liquid separator is to pump a freshly prepared 1% w/v sodium thiosulfate solution through the system for 5-10 minutes. You must then remove the thiosulfate by pumping distilled water through the system for 5-10 minutes.

Replacement Procedures

Pump Tubes

To remove the pump tubes, use the reverse procedure of that given on Page 27.

Reaction Coil

To replace the reaction coil:

1 Remove the gas/liquid separator as described previously.

2 Remove the thumbscrew holding the reagent manifold cover in place. Remove the cover by pulling the top of the cover away from the manifold block and pushing the cover down.

3 Remove the black fluoro-elastomer sleeve from the end of the reaction coil. Pull the end of the reaction coil through the hole in the manifold block, then pull the reaction coil out of the channel.

4 Slide the 'T' connection and the pump tubing connector out of their slots. You will find it easier if you remove all of the 'T' pieces and pump connectors.

5 Hold the upper 'T' piece and sleeve firmly with one hand and pull the reaction coil out of the sleeve.

6 Fit the new reaction coil into the sleeve, then replace all of the pump tube connections and ‘T’ connections into their slots.

7 Wind the reaction coil around the channels and out through the hole in the manifold block. Ensure that there are no kinks in the tubing.

8 Fit the black fluoro-elastomer sleeve onto the end of the reaction coil.

9 Replace the manifold cover.

10 Replace the separator.

Uptake Capillary Tubes

To replace an uptake capillary tube:

1 Remove the separator and manifold cover as described previously.

2 Slide the pump tubing connector out of its slot.

3 Pull the capillary out of its sleeve.

4 Push the end of the new capillary into its sleeve.

5 Replace the connector into its slot.

6 Replace the cover.

Fuses

FS1, FS2: T0.2 AH 250 V, IEC60127-2 Sheet 5, 5x20 mm, Littelfuse 0215200 or equivalent

Fuse information on the rear of the instrument is the most up to date.

NOTE For safety reasons, any other internal fuse or circuit breaker is not operator-accessible, and should only be replaced by Agilent authorized representatives.

WARNING

Shock Hazards

To prevent reduced safety protection or unwanted fusing always ensure that the marking on the fuse matches the screening shown adjacent to the voltage selector switches.

Check the fuses as follows:

1. Disconnect the unit from the mains power supply.
2. Undo the fuse cap by pressing the cap and turning it counter-clockwise.
3. Pull the cap out carefully. The fuse should be held in the fuseholder in the fuse cap.
4. Check that the fuse is the correct type and that it is not damaged. If necessary, replace the fuse in the holder.
5. Place the fuse into the cap, push the cap in, and turn the cap clockwise.
6. Reconnect the unit to the mains power supply.

NOTE If a fuse blows repeatedly, it may indicate other problems with the accessory. You should contact your local Agilent sales office and arrange a service call.

Spare Parts

NOTE Use only Agilent supplied parts unless otherwise indicated.

The following spare parts are available from Agilent for your VGA 77:

Module for AA includes reagent module, tubing and connectors	9910062100
Module for ICP includes module, tubing and connectors	9910062200
Absorption cell for AA. 2 cells, separator to cell tubing (fluoro-elastomer)	9910040000
Flow through mercury cell kit. Includes cell and separator to cell tubing (fluoro-elastomer)	9910040700
Cell holder for Mark 7 burner	0110654990
Cell holder mounting kit for Mark VI burner	0110375990
Cell holder for Mark V burner mount	0110317790
Cell holder mounting kit for Mark V burner	0410117300
Tubing kit for AA	9910061900
Tubing kit for ICP	9910061800
Gas/liquid separator kit for AA includes separator and rubber mounting strap	9910071100
Gas/liquid separator kit for ICP includes separator, tubing and rubber mounting strap	9910056900
Fuse, T0.2AH0250V IEC sheet 5, 5 x 20 mm (0.8 in.)	1910009300
Pump tubes, black/black (pack of 12)	3710027200
Pump tubes, purple/black (pack of 12)	3710027300
Polyethylene capillary tubing 1 m (3.3 ft) lengths.	2410020500
Fluoro-elastomer tubing, small. 1 m (3.3 ft) lengths.	3710026800
Fluoro-elastomer tubing, AA. 0.66 m (2.2 ft) lengths, 2.8 mm (0.110 in.) ID.	3710026390
Drain tubing from gas-liquid separator (2 meter length required) 2 m (6.6 ft) length, 10 mm (0.4 in.) ID.	3710009200
Reagent vessel, 500 mL	6610011600
Replacement drip tray	7010017200

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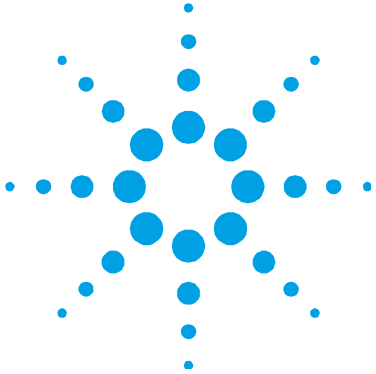

7. Appendix – AA Performance Log

Date	Operator	Uptake rate	Element	Concentration	Abs

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In This Book

The manual describes the following:

- Introduction
- Site Requirements and Specifications
- Hardware Installation
- Operation of Hardware and Software
- Analytical Notes
- Laboratory Procedures
- Maintenance Procedures
- Instrument Cleaning
- Spare Parts
- Performance Log

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